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### (54) DISPERSION STABILIZER FOR SUSPENSION POLYMERIZATION OF VINYL COMPOUND

#### (57)Abstract:

PURPOSE: To obtain a dispersion stabilizer which exhibits low foaming properties in the suspension polymn. of a vinyl compd. and is excellent in capability of producing uniform and porous vinyl polymer particles.

CONSTITUTION: This stabilizer comprises a polyvinyl alcohol of which the absorbance at 280nm (A) in an ultraviolet absorption spectrum of a 0.1wt.% aq. soln. is 0.1 or higher, the absorbance at 320nm (B) is 0.03 or higher, and the ratio of absorbance A to absorbance B is lower than 0.3 and in which the block character of residual acetic acid groups is 0.4 or higher.

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the distributed stabilizer for suspension polymerizations of a vinyl system compound. It is related with the distributed stabilizer for suspension polymerizations of vinyl system compounds, such as a vinyl chloride which was remarkably [ it is still more detailed and ] excellent in the defoaming effectiveness.

[0002]

[Description of the Prior Art] When manufacturing vinyl system polymers, such as vinyl chloride system resin, industrially, vinyl system compounds, such as a vinyl chloride, are distributed under existence of a distributed stabilizer in an aqueous medium, and the suspension polymerization which performs a polymerization using an oil solubility catalyst is carried out widely. Generally, as a factor which governs the quality of a vinyl system polymer, although the class of conversion, a water-monomer ratio, polymerization temperature, the class of catalyst and an amount, the type of a polymerization tank, an agitating speed, or distributed stabilizer etc. is mentioned, the effect by the class of distributed stabilizer is dramatically large especially.

[0003] As engine performance required of the distributed stabilizer for suspension polymerizations of a vinyl system compound \*\* There is work which makes Sharp particle size distribution of the vinyl system polymer particle obtained as much as possible, \*\* In order to enlarge rate of absorption of a plasticizer, to make workability easy, and to make easy clearance of monomers, such as a vinyl chloride which remains in a polymer particle, and to prevent generation of the fish eye in mold goods etc. It is mentioned that there is work which makes each polymer particle porosity, that there is work which forms a polymer particle with large \*\* restoration specific gravity, etc. conventionally, as a distributed stabilizer for suspension polymerizations of a vinyl system compound, a cellulosic or partial saponification polyvinyl alcohol, such as methyl cellulose and a carboxymethyl cellulose, etc. is independent -- or it is combined and used. However, the conventional distributed stabilizer had the problem that the military requirement of the above-mentioned \*\* - \*\* was not filled.

[0004] The suspension polymerization of vinyl system compounds, such as a vinyl chloride, has a common approach of preparing an aqueous medium, a distributed stabilizer, a polymerization initiator, a vinyl system compound, etc. into curing units, usually being carried out by the batch type, carrying out temperature up, after adding the additive needed further, and making a polymerization reaction perform. Recently, in order to raise productivity, shortening the time amount which polymerization 1 batch takes is called for, a reflux condenser etc. is installed in the suspension polymerization of a vinyl system compound, the cooling effectiveness of the heat of polymerization is raised, or the approach of shortening a heating up time by the approach (the hot charging method) of preparing the aqueous medium heated beforehand is used. However, since foaming under polymerization was intense, the sensitive volume in curing units decreased, and when the distributed stabilizer for suspension polymerizations of the conventional vinyl system compound was used, when, or a temperature control becomes impossible when curing units with a reflux condenser were used, or the hot charging method

was used, there was a fatal fault that the porosity of a vinyl system polymer particle fell. [ productivity ]  
On the other hand, in order to prevent foaming, when the defoaming agent etc. was added, there was a problem that the thermal stability of the vinyl system polymer particle to generate fell.

[0005] A degree of polymerization 2000, saponification degree % of the polyvinyl alcohol and of 80 mols, degrees of polymerization 700-800, and saponification degree % of the polyvinyl alcohol of 70 mols are indicated as a distributed stabilizer for suspension polymerizations of a vinyl chloride by poval (publishing office: a giant-molecule publication meeting, 1984) [well-known reference (a)]. However, the satisfactory engine performance was not obtained to the demand of the above-mentioned \*\* - \*\*, but the distributed stabilizer indicated by well-known reference (a) had the fault that the fizz under polymerization was intense. In JP,5-88251,B [well-known reference (b)], the ratio of a weight average degree of polymerization and a number average degree of polymerization is 3.0 or less or more in 500, and average degree of polymerization has a carbonyl group in intramolecular, and this is adjoined, it has a vinylene radical, and the distributed stabilizer for suspension polymerizations of the vinyl compound with which the absorbance of 280nm and 320nm by the ultraviolet absorption spectrum of a water solution consists of polyvinyl alcohol whose ratio with an absorbance of 320nm to 280nm is 0.30 or more in 0.3 or more and 0.15 or more 0.1%, respectively be indicated. However, the distributed stabilizer indicated by well-known reference (b) had the problem that the fizz in curing units was intense.

[0006]

[Problem(s) to be Solved by the Invention] The approach of controlling polymerization temperature with the jacket or coil in the approach (the cold charging method) of preparing the aqueous medium of the ordinary temperature whose object of this invention is the conventional suspension-polymerization approach of a common vinyl system compound in curing units, and curing units from the first It is in offering the distributed stabilizer which the defoaming effectiveness in curing units is remarkably excellent in, and fulfills the demand characteristics of the aforementioned \*\* - \*\* simultaneously also in the approach of using curing units with a capacitor, the hot charging method, and the hot charging method using curing units with a capacitor.

[0007]

[Means for Solving the Problem] In order that this invention persons may solve the above-mentioned technical problem, as a result of repeating examination wholeheartedly, the absorbance (a) of 280nm by the ultraviolet absorption spectrum of the 0.1 % of the weight water solution of concentration is size from 0.1. The absorbance (b) of 320nm by the ultraviolet absorption spectrum of this water solution is 0.03 or more. It came to complete a header and this invention for the distributed stabilizer for suspension polymerizations of the vinyl system compound which consists of a polyvinyl alcohol system polymer (an absorbance b) / whose absorbance (a) is less than 0.3 and, whose block character of a residual acetic-acid radical is 0.4 or more.

[0008] It explains in more detail about this invention. It is 60-88-mol %, and 65-75-mol% of the range where the saponification degree of the polyvinyl alcohol system polymer of this invention is desirable is more desirable, and is still more desirable. [ 68-74-mol% of ] It is 500-1500, as for the range where the viscosity average polymerization degree (it is hereafter written as average degree of polymerization) of a polyvinyl alcohol system polymer is desirable, 600-1200 are more desirable, and 680-900 are still more desirable.

[0009] The absorbance (a) of 280nm by the ultraviolet absorption spectrum of the 0.1 % of the weight water solution of concentration is size, 0.2 or more are more desirable than 0.1, and, as for the polyvinyl alcohol system polymer of this invention, it is still more desirable that it is 0.25 or more. The absorbance (b) of 320nm by the ultraviolet absorption spectrum of this water solution is 0.03 or more, and it is desirable that it is 0.05-0.2. It is less than 0.3, as for (an absorbance b) / absorbance (a), 0.25 or less are desirable, and 0.2 or less are still more desirable.

[0010] It is 0.4 or more, as for the block character of the residual acetic-acid radical of the polyvinyl alcohol system polymer of this invention, 0.43 or more are desirable, and 0.5 or more are still more desirable. About the block character of a residual acetic-acid radical, the measuring method etc. is explained by poval (publishing office: a macromolecule publication meeting, 1984) and

Macromolecules, and 10,532 (1977) in full detail here.

[0011] Although there is especially no limit in the manufacture approach of the polyvinyl alcohol system polymer of this invention, in case the radical polymerization of the vinyl ester monomer is carried out, the method of carrying out a polymerization to intramoleculars, such as an acetaldehyde and a butyraldehyde, under coexistence of the compound which has a carbonyl group, processing the obtained polymer from acids, such as alkali, such as a sodium hydroxide and ammonia, or a hydrochloric acid, and Para toluenesulfonic acid, and saponifying a vinyl ester polymer is simple, and efficient. The approach of making the block character of the residual acetic-acid radical of the polyvinyl alcohol system polymer of this invention 0.4 or more is acquired by carrying out acid saponification of the polyvinyl ester of a raw material. Even when carrying out alkali saponification of the polyvinyl ester of a raw material, it is obtained by heat-treating the obtained polyvinyl alcohol system polymer. In this case, before heat-treating the obtained polyvinyl alcohol system polymer, after organic solvents', such as an acetone's, a methanol's, and methyl acetate's, washing a polyvinyl alcohol system polymer and carrying out the content of salts, such as sodium acetate, to 0.5 or less % of the weight preferably 0.7 or less % of the weight to a polyvinyl alcohol system polymer, the method of performing heat treatment is suitable. As vinyl ester at the time of carrying out the radical polymerization of the vinyl ester monomer, formic-acid vinyl, vinyl acetate, propionic-acid vinyl, butanoic acid vinyl, isobutyric-acid vinyl, vinyl pivalate, BASA tic acid vinyl, caproic-acid vinyl, caprylic-acid vinyl, caprylic-acid vinyl, lauryl acid vinyl, palmitic-acid vinyl, stearin acid vinyl, oleic acid vinyl, etc. are mentioned, for example. the polyvinyl alcohol system polymer used for this invention -- ion radicals, such as ammonium, a carboxyl group, and a sulfone radical, the Nonion radical, or (long-chain) an alkyl group -- less than [ 10 mol % ] -- you may introduce. The saponification degree in this case is called for from a vinyl ester radical and a vinyl alcohol radical, and the introduced ion radical is not contained.

[0012] Although the engine performance which was excellent also when the polyvinyl alcohol system polymer (A) of this invention was independently used as a distributed stabilizer is demonstrated, when weight ratio [ of a component (A) and a component (B) ] (A)/(B) is 40 / 60 - 95/5, the more excellent engine performance is demonstrated in a concomitant use system with the polyvinyl ester system polymer below saponification degree 60 mol % (B).

[0013] It is less than [ 60 mol % ], and 20 - 55-mol% of the saponification degree of a polyvinyl ester system polymer (B) is desirable, and is further more desirable. [ 25 - 45 mol% of ] They is 40 / 60 - 95/5, as for weight ratio [ of a component (A) and a component (B) ] (A)/(B), 50 / 50 - 90/10 are more desirable, and 60 / 40 - 80/20 are further more desirable. When larger than 95/5, a porosity improvement effect may not be seen for weight ratio [ of a component (A) and a component (B) ] (A)/(B), and the stability of a polymerization may be lost when it is less than 40/60. Although there is especially no limit about the polymerization degree of a polyvinyl ester system polymer (B), 1000 or less are desirable, 100-550 are more desirable, and 200-400 are further more desirable.

[0014] A polyvinyl ester system polymer (B) is water-insoluble nature or water-dispersion, and self-emulsifiability could be given by introducing an ion radical etc. In this invention, the gestalt with which the polyvinyl ester system polymer (B) was used together by the distributed stabilizer of the polyvinyl alcohol system polymer of this invention as a distributed assistant is also called dispersant stabilizer.

[0015] There is especially no limit in the manufacture approach of a polyvinyl ester system polymer (B), and a well-known thing is used suitably conventionally. for example, the polyvinyl ester system polymer which has an ion radical in the side chain indicated by JP,1-95103,A, the polyvinyl ester system polymer which has an ionicity radical at the end indicated by WO 91/15518, the well-known Nonion radical, or (long-chain) an alkyl group -- less than [ 10 mol % ] -- the polyvinyl ester system polymer which it has is used suitably.

[0016] Next, the manufacture approach of the vinyl system polymer by the suspension polymerization of the vinyl system compound using the distributed stabilizer of this invention is explained. Especially a limit does not have the temperature of the aquosity medium used in the manufacture approach of a vinyl system polymer, and not only about 20-degree C chilled water but also warm water 90 degrees C or more is used suitably. The aquosity medium containing the water solution or other organic solvents

containing various kinds of addition components other than pure water can mention the medium which constitutes this heating aqueous medium. Moreover, the amount of supply at the time of teaching a heating aqueous medium to a polymerization reaction system should just be an amount which can fully warm a polymerization reaction system. Moreover, in order to raise cooling effectiveness, curing units with a reflux condenser are also used suitably. In the manufacture approach of a vinyl system polymer, although especially a limit does not have the amount of the distributed stabilizer used, it is usually below 5 weight sections to the vinyl system compound 100 weight section, and 0.01 - 1 weight section is desirable, and the 0.02 - 0.2 weight section is further more desirable. Saponification degree % of the polyvinyl alcohol of 65-98 mols usually used in case the suspension polymerization of the vinyl system compounds, such as a vinyl chloride, is carried out in an aqueous medium although the distributed stabilizer of this invention may be used independently, Methyl cellulose, hydroxyethyl cellulose, hydroxypropylcellulose, Water-soluble cellulose ether, such as hydroxypropyl methylcellulose, Water-soluble polymers, such as gelatin, sorbitan monolaurate, a sorbitan trio rate, Oil solubility emulsifiers, such as glycerol tristearate and an ethylene oxide propylene oxide block copolymer, Water-soluble emulsifiers, such as polyoxyethylene sorbitan monolaurate, polyoxyethylene glycerol olate, and lauric acid sodium, etc. may be used together. Although there is especially no limit about the addition, per [ 0.01 ] vinyl system compound 100 weight sections, such as a vinyl chloride, - the 1.0 weight sections are desirable.

[0017] In addition, various additives can also be added if needed. As various additives, polymerization inhibitor, such as polymerization-degree modifiers, such as an acetaldehyde, a butyraldehyde, a trichloroethylene, perchloroethylene, or mercaptans, a phenolic compound, a sulfur compound, and N-oxide compound, etc. is mentioned, for example. Moreover, even if it is also arbitrary to add pH regulator, a scale inhibitor, a cross linking agent, etc. and it uses together two or more above-mentioned additives, it does not interfere. On the other hand, a polymerization initiator is also easy to be what is conventionally used for the polymerization of vinyl system compounds, such as a vinyl chloride. To this, for example, diisopropyl peroxy dicarbonate, di-2-ethylhexylperoxycarbonate, Par carbonate compounds, such as diethoxy ethyl peroxy dicarbonate, T-butyl peroxy neodecanate, alpha-cumyl peroxy neodecanate, Par ester compounds, such as t-butyl peroxy neodecanate, acetyl cyclohexyl sulfonyl peroxide, Peroxides, such as 2, 4, and 4-trimethyl pentyl-2-peroxy phenoxy acetate, 2,2'-azobis isobutyronitrile, azobis-2,4-dimethylvaleronitrile, Azo compounds, such as azobis (4-methoxy-2,4-dimethylvaleronitrile), etc. are mentioned, and it can also be further used for these combining potassium persulfate, ammonium persulfate, a hydrogen peroxide, etc.

[0018] As a vinyl system compound which can carry out a suspension polymerization using the distributed stabilizer of this invention As a comonomer by which the monomer mixture (50 % of the weight or more of vinyl chlorides) which specifically makes a subject a vinyl chloride besides a vinyl chloride independent is included, and copolymerization is carried out to this vinyl chloride Vinyl ester, such as vinyl acetate and propionic-acid vinyl, a methyl acrylate (meta), (Meta) Olefins, such as acrylic ester (meta), such as an ethyl acrylate, ethylene, and a propylene, a maleic anhydride, acrylonitrile, an itaconic acid, styrene, a vinylidene chloride, vinyl ether, other vinyl chlorides, and the monomer that can be copolymerized are illustrated. Furthermore, the distributed stabilizer of this invention can be used in homopolymerization and copolymerization of the above-mentioned vinyl system compound which do not contain a vinyl chloride. What is necessary is just to define the preparation rate of each component, polymerization temperature, etc. according to the conditions conventionally adopted by the suspension polymerization of vinyl system compounds, such as a vinyl chloride, in carrying out a suspension polymerization using the distributed stabilizer of this invention. Moreover, about the preparation sequence or the ratio of a vinyl system compound, a polymerization initiator, a distributed stabilizer, a heating aqueous medium, and other additives, it is not restricted at all. Moreover, before teaching a vinyl system compound to curing units, the method of heating the vinyl system compound is also suitably used, at the same time it uses warm water.

[0019]

[Example] Although an example is given below and this invention is explained in more detail, this

invention is not limited at all by these examples. In addition, unless "%" and the "section" have a notice especially in the following examples, "% of the weight" and the "weight section" are meant.

[0020] After fully carrying out the nitrogen purge of the interior for the example [polyvinyl alcohol system polymer] of manufacture vinyl acetate (it outlines Following VAc) 5235 section, the methanol 380 section, and the acetaldehyde 115 section for a reaction container, the outside temperature was raised to 65 degrees C, and the methanol solution 20 section which contains the 2,2'-azobis isobutyronitrile 1 section which carried out the nitrogen purge beforehand in the place where the internal temperature amounted to 60 degrees C was added. The conversion of 5 hours after was 60.7%. The container was cooled 5 hours after, actuation of driving out with a methanol VAc which remains under reduced pressure out of a system was performed, adding a methanol, and the methanol solution of polyvinyl ester (it outlines Following PVAc) was obtained (55% of concentration). After taking a part of this methanol solution, performing saponification by 30% of PVAc concentration, 2% of water content, and  $[\text{NaOH}]/[\text{VAc}]=0.05$  (mole ratio) and SOKKURE washing by the methanol refining the obtained polyvinyl alcohol (PVA), when the saponification degree was measured, it was 99.2-mol %. Moreover, it was 780, when limiting viscosity was measured at 30 degrees C of underwater and average degree of polymerization was calculated. Next, the methanol solution of NaOH was added and it saponified at 40 degrees C by 20% of PVAc concentration, 1% of water content, and the conditions of 30% of methyl acetate so that some above-mentioned methanol solutions might be taken, water, a methanol, and methyl acetate might be added and it might be set to  $[\text{NaOH}]/[\text{VAc}]=0.02$  (mole ratio). After [ of alkali addition ] 5 minutes, since the system gelled, the mixer ground, the mixed liquor of methyl acetate / water = 8/2 was heated at a saponification system, tales-doses \*\*\*\*, and 60 degrees C, and the reaction was continued for further 1 hour. The saponification object (PVA) was filtered after that, it dried at 60 degrees C among the air forced oven, and PVA was obtained. It refined by performing SOKKURE washing according obtained PVA to a methanol for 10 hours. The sodium acetate content measured with isotachophoresis (iso octopus FORESHISU) was 0.48%. Then, it heat-treated for 4 hours in a 150 degree C air forced oven and under the air ambient atmosphere. When the saponification degree was measured, it was 72-mol %. moreover, the absorbance of 280nm and 320nm by the ultraviolet absorption spectrum of 0.1% water solution -- respectively -- 0. -- it was 45 and 0.08. Moreover, the block character of the residual acetic-acid radical measured using C13-NMR was 0.46. The polyvinyl alcohol system polymer as shown in a table 1 was prepared by changing charges, such as VAc, a methanol, and an acetaldehyde, suitably, and changing suitably the temperature of heat treatment in an air forced oven, and time amount, using alkali or an acid as a saponification catalyst.

[0021] [Polymerization of a vinyl chloride] The 70% toluene solution 0.04 of the deionized water 1 section which melted the distributed stabilizer shown in a table 1 (examples 1-11, examples 1-2 of a comparison), and diisopropyl peroxy dicarbonate section was taught to the autoclave made from glass lining with a reflux condenser, and after having deaerated the inside of an autoclave until it was set to 50mmHg(s), and removing oxygen, the warm water 39 80-degree C section and the vinylchloride monomer 30 section were simultaneously taught under churning. The oil level in the event of preparation being completed was 60% of height from the base of curing units, and the internal temperature was 50 degrees C. 50 degrees C of internal temperatures were maintained after that, and the polymerization was continued. Although 7.0kg/cm<sup>2</sup> of pressures in an autoclave was G at the time of polymerization initiation, when 4.0kg/cm<sup>2</sup> was set to G 6 hours after polymerization initiation, the polymerization was suspended, the unreacted vinylchloride monomer was purged, and ejection dehydration desiccation of the contents was carried out. The following approach estimates the engine performance of the obtained vinyl chloride resin, and the result is shown in a table 2. The polymerization yield of a vinyl chloride polymer was 85%, and average degree of polymerization was 1300.

[0022] (1) Time amount was made into plasticizer absorptivity (minute) until it added 200g of dioctyl phthalates to this and torque descended to the planetary mixer connected to plasticizer absorptivity plastograph from the time of addition, after having put in 400g of obtained vinyl chloride polymer powder, preheating, agitating by 60rpm (4 minutes) and considering as 88 degrees C.

(2) From the approach indicated by CPA(Cold Plasticizer Absorption: cold plasticizer absorbed amount) ASTM-D 3367-75, the absorbed amount of the dioctyl phthalate in 23 degrees C was measured.

(3) Observe the foaming condition in curing units by viewing at the time of fizz assessment polymerization termination, and the following notations show it at it.

O : -foaming-less O : the bubble was accepted even in 62 - 65% of height from the base of curing units.

\*\* : The bubble was accepted even in 66 - 70% of height from the base of curing units.

\*\* : The bubble was accepted even in 90 - 100% of height from the base of curing units.

x: The bubble was accepted even in 100% of height from the base of curing units, and the bubble was further got blocked in the reflux condenser.

[0023]

[A table 1]

	ポリビニルアルコール(A)						ポリビニルエステル(B)			配 合 比 (A)/(B)	3) 使用量 (部)
	重合度	けん化度 (モル%)	1) D <sub>280</sub>	2) D <sub>320</sub>	D <sub>320</sub> /D <sub>280</sub>	ブ ロ ッ ク キ ャ ラ ク タ ー	重合度	けん化度 (モル%)	変性基		
実施例 1	1480	87	0.15	0.042	0.28	0.40				10/0	0.1
" 2	780	80	0.2	0.050	0.25	0.41				10/0	"
" 3	650	72	0.25	0.055	0.22	0.43				10/0	"
" 4	1170	76	0.3	0.057	0.19	0.43				10/0	"
" 5	720	72	0.45	0.081	0.18	0.46				10/0	"
" 6	700	70	0.78	0.109	0.14	0.52				10/0	"
" 7	920	68	0.60	0.168	0.28	0.80				10/0	"
" 8	720	65	0.45	0.081	0.18	0.46	230	35	—	7/3	0.12
" 9	720	65	0.45	0.81	0.18	0.46	230	42	イタコン酸0.5モル	7/3	"
" 10	720	65	0.45	0.81	0.18	0.46	480	46	末端カルボキシ基	7/3	"
" 11	720	65	0.45	0.81	0.18	0.46	350	55	—	7/3	"
比較例 1	700	70	0.08	0.02	0.25	0.42				10/0	0.1
" 2	720	76	0.60	0.21	0.35	0.38				10/0	"

1) D<sub>280</sub> : 280nmにおける吸光度(1%水溶液)

2) D<sub>320</sub> : 320nmにおける吸光度( " )

3) 使用量: 塩化ビニルモノマー100部当たりの使用量

[0024]

[A table 2]



	重合安定性	C P A (%)	可塑剂吸収性 (分)	発泡状態
実施例 1	良好	30	3.0	△
" 2	"	31	2.6	○
" 3	"	32	2.7	○
" 4	"	34	2.4	◎
" 5	"	36	2.3	◎
" 6	"	35	2.4	◎
" 7	"	31	2.8	○
" 8	"	38	2.0	◎
" 9	"	37	2.1	◎
" 10	"	38	2.3	◎
" 11	"	36	2.5	◎
比較例 1	不良	21	5.0	×
" 2	"	28	3.5	▲

[0025]

[Effect of the Invention] The distributed stabilizer of this invention is compared with the conventional distributed stabilizer, since the foaming behavior under polymerization is very small, the sensitive volume in curing units increases, its productivity improves, and it becomes easy [ the temperature control of curing units ] in the suspension polymerization using curing units with a reflux condenser, the suspension polymerization by the hot charging method, or the suspension polymerization by the hot charging method using curing units with a reflux condenser. Moreover, when the distributed stabilizer for suspension polymerizations of this invention is used, it is, lump nature is good, the porosity of a vinyl system polymer particle improves, particle diameter is large, the distribution is sharp, and there is little scattering at the time of handling, and assessment industrial from the high thing of restoration specific gravity to go away to a making machine etc. moreover is plasticizer rate of absorption is large and very high [ the obtained vinyl system polymer particle ].

[Translation done.]